



October 26, 2015

Tom Moe USS Corporation P.O. Box 417 Mountain Iron, MN 55768

RE: Project: NPDES Line 3 Wkly Pace Project No.: 1255278

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on October 14, 2015. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Heather R Zika

Haller Zto

heather.zika@pacelabs.com

Project Manager

Enclosures

cc: Terri Sabetti, Northeast Technical





315 Chestnut Street Virginia, MN 55792 (218) 742-1042



CERTIFICATIONS

Project: NPDES Line 3 Wkly

Pace Project No.: 1255278

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792
Alaska Certification #MN01084
Arizona Department of Health Certification #AZ0785
Minnesota Dept of Health Certification #: 027-137-445

North Dakota Certification: # R-203

Wisconsin DNR Certification # : 998027470 WA Department of Ecology Lab ID# C1007 Nevada DNR #MN010842015-1 Oklahoma Department of Environmental Quality

315 Chestnut Street Virginia, MN 55792 (218) 742-1042



SAMPLE SUMMARY

Project: NPDES Line 3 Wkly

Pace Project No.: 1255278

| Lab ID | Sample ID | Matrix | Date Collected | Date Received | |
|------------|--------------------------|--------|----------------|----------------|--|
| 1255278001 | WS002 Scrubber Makeup | Water | 10/14/15 08:45 | 10/14/15 15:10 | |
| 1255278002 | WS003 Thickener Overflow | Water | 10/14/15 08:45 | 10/14/15 15:10 | |



SAMPLE ANALYTE COUNT

Project: NPDES Line 3 Wkly

Pace Project No.: 1255278

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|------------|--------------------------|-----------|----------|----------------------|------------|
| 1255278001 | WS002 Scrubber Makeup | EPA 200.7 | MAR | 3 | PASI-V |
| | | EPA 300.0 | DMB | 1 | PASI-V |
| 1255278002 | WS003 Thickener Overflow | EPA 200.7 | MAR | 3 | PASI-V |
| | | EPA 300.0 | DMB | 1 | PASI-V |



ANALYTICAL RESULTS

Project: NPDES Line 3 Wkly

Pace Project No.: 1255278

Date: 10/26/2015 04:54 PM

| Sample: WS002 Scrubber Makeup | Lab ID: | 1255278001 | Collected | : 10/14/15 | 08:45 | Received: 10/ | 14/15 15:10 Ma | atrix: Water | | |
|---|-----------------------------------|-------------------------------|---|----------------------------|---------------------|---|----------------------------------|--------------|------|--|
| | | | Report | | | | | | | |
| Parameters | Results | Units | Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual | |
| 200.7 MET ICP, Lab Filtered | Analytical | Method: EPA | 200.7 Prepar | ation Meth | od: EP/ | A 200.7 | | | | |
| Calcium, Dissolved | 87.3 | mg/L | 5.0 | 0.29 | 10 | 10/19/15 11:30 | 10/20/15 13:09 | 7440-70-2 | | |
| Magnesium, Dissolved | 190 | mg/L | 5.0 | 0.67 | 10 | 10/19/15 11:30 | 10/20/15 13:09 | 7439-95-4 | | |
| Total Hardness, Dissolved | 1000 | mg/L | 100 | 50.0 | 10 | 10/19/15 11:30 | 10/20/15 13:09 | | | |
| 300.0 IC Anions 28 Days | Analytical | Method: EPA | 300.0 | | | | | | | |
| Sulfate | 724 | mg/L | 40.0 | 1.8 | 20 | | 10/22/15 12:11 | 14808-79-8 | | |
| Sample: WS003 Thickener Overflo | w Lab ID: | 1255278002 | Collected | : 10/14/15 | 08:45 | Received: 10/ | 14/15 15:10 Ma | atrix: Water | | |
| | | | | | | | | | | |
| | | | Report | | | | | | | |
| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual | |
| | | Units Method: EPA 2 | Limit | | | · | Analyzed | CAS No. | Qual | |
| Parameters | | | Limit | | | · | Analyzed 10/20/15 13:12 | | Qual | |
| 200.7 MET ICP, Lab Filtered Calcium, Dissolved | Analytical | Method: EPA 2 | Limit | ation Meth | od: EP/ | A 200.7 | | 7440-70-2 | Qual | |
| 200.7 MET ICP, Lab Filtered Calcium, Dissolved Magnesium, Dissolved | Analytical | Method: EPA 2 | Limit — — — — — — — — — — — — — — — — — — — | ation Meth | od: EP/ | A 200.7 10/19/15 11:30 | 10/20/15 13:12 | 7440-70-2 | Qual | |
| 200.7 MET ICP, Lab Filtered | Analytical 724 33.2 1940 | Method: EPA 2 mg/L mg/L | Limit | ation Meth 0.29 0.67 | od: EP/ 10 10 | A 200.7 10/19/15 11:30 10/19/15 11:30 | 10/20/15 13:12 10/20/15 13:12 | 7440-70-2 | Qual | |



QUALITY CONTROL DATA

Project: NPDES Line 3 Wkly

Pace Project No.: 1255278

Date: 10/26/2015 04:54 PM

QC Batch: MPRP/6015 Analysis Method: EPA 200.7

QC Batch Method: EPA 200.7 Analysis Description: 200.7 MET Dissolved

Associated Lab Samples: 1255278001, 1255278002

METHOD BLANK: 259013 Matrix: Water

Associated Lab Samples: 1255278001, 1255278002

Reporting Blank Parameter MDL Result Limit Qualifiers Units Analyzed Calcium, Dissolved ND 0.50 10/20/15 11:53 mg/L 0.029 Magnesium, Dissolved mg/L ND 0.50 0.067 10/20/15 11:53

LABORATORY CONTROL SAMPLE: 259014 Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Calcium, Dissolved 50 49.6 99 85-115 mg/L

 Calcium, Dissolved
 mg/L
 50
 49.6
 99
 85-115

 Magnesium, Dissolved
 mg/L
 50
 49.8
 100
 85-115

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 259015 259016 MSD MS 1255207001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits **RPD** RPD Qual Calcium, Dissolved mg/L 8.2 50 50 57.9 58.1 99 100 70-130 0 20 Magnesium, Dissolved mg/L 2.0 50 50 51.7 51.4 100 99 70-130 20

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 259017 259018 MS MSD MS 1255322001 MSD MS Spike Spike MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits RPD RPD Qual Calcium, Dissolved 50 2 10.0 50 59.0 60.1 98 70-130 20 mg/L 100 50 2 Magnesium, Dissolved 1.0 50 49.3 50.3 97 99 70-130 20 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALITY CONTROL DATA

Project: NPDES Line 3 Wkly

Pace Project No.: 1255278

Date: 10/26/2015 04:54 PM

QC Batch: WETA/14293 Analysis Method: EPA 300.0

QC Batch Method: EPA 300.0 Analysis Description: 300.0 IC Anions

Associated Lab Samples: 1255278001, 1255278002

METHOD BLANK: 260189 Matrix: Water

Associated Lab Samples: 1255278001, 1255278002

ParameterUnitsBlank Reporting ResultReporting LimitMDLAnalyzedQualifiersSulfatemg/LND2.00.08910/22/15 05:20

LABORATORY CONTROL SAMPLE: 260190

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Sulfate mg/L 50 49.6 99 90-110

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 260191 260192 MS MSD

1255466001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits RPD RPD Qual Sulfate 90-110 0 20 mg/L 9.4 50 50 59.8 59.8 101 101

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 260193 260194

MS MSD MS MS MSD 1255277001 Spike Spike MSD % Rec Max Result Limits RPD Parameter Units Conc. Conc. Result Result % Rec % Rec RPD Qual Sulfate 185 100 100 284 285 100 100 90-110 0 20 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALIFIERS

Project: NPDES Line 3 Wkly

Pace Project No.: 1255278

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

Date: 10/26/2015 04:54 PM

PASI-V Pace Analytical Services - Virginia



QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: NPDES Line 3 Wkly

Pace Project No.: 1255278

Date: 10/26/2015 04:54 PM

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|--------------------------|---|------------------------|--------------------------|------------------------|----------------------|
| 1255278001 1255278002 | WS002 Scrubber Makeup WS003 Thickener Overflow | EPA 200.7 EPA 200.7 | MPRP/6015 MPRP/6015 | EPA 200.7 EPA 200.7 | ICP/4665 ICP/4665 |
| 1255278001 1255278002 | WS002 Scrubber Makeup WS003 Thickener Overflow | EPA 300.0 EPA 300.0 | WETA/14293 WETA/14293 | | |

| | | | | | | | 23 | # | 8 | 60 | œ | 7 | o · | Ó | 4 | ۵ | N | - | ITEM# | Requeste | Phone | Disagna. | Email: | Address: | Company: | xequire | Section A | |
|---|---|----------|---|---|------------|----------------------------|----------|---|---|----------|---|---|-----|---|----|---|------------------------------|-----------------------------------|--|--|---------------------------|---|------------------------------|---------------|---------------|-------------------------|------------|--|
| | | | | | | ADDITIONAL COMMENTS | | | | | | | | | | | WS-003 Thickner Overflow | WS-002 Scrubber Make-Up | SAMPLE ID One Character per box. (A-Z, 0-91,-) Sample ids must be unique | Requested Due Date: | Fax | lear | Mt. Iron, MN 55768 Email: | : | | 15 | A | Pace Analytical me administra |
| | | | | | So- | | | | | | | | | | | | WT | WT | MATRIX CODE (see valid codes to left) | Project #: | Project Name: | Draine Name: | Purchase Order #: | Copy To: | 1 | Required Project | Section B | |
| | | | | | Carlamans | NOUISHE | | | | | | | | | | | | | SAMPLE TYPE (G=GRAB C=COMP) | | NPDE | | # | | Tom Moe | ct Inform | ; I | |
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| TEMP | o in C | | | | 02 | 1 | L | | | | l | | | | | | | <u> </u> | Residual Chlorine (Y/N) | | SIZ | | Regu | | 100,000 | ige : | vaste ei | e euro es dem |
| (Y/N) Custo Sealed | ody d | | | | 2 | SAMPLE CONDITIONS | | | | | | | | | | | ĽĒ,ĽĒ | LF,LF | Residual Chiorina (Y/N) | A STATE OF THE PARTY OF THE PAR | State / Location | | Regulatory Agency | | | | | |
| Coole (Y/N) Samp Intact (Y/N) | oles | | | | | JONES | | | | | | | | | ١. | | | | | TO SECTION OF THE PARTY. | | | | | | f | | |

Pace Analytical*

hold, incorrect preservative, out of temp, incorrect containers)

Document Name: Sample Condition Upon Receipt Form

Document No.: F-VM-C-001-Rev.06

Document Revised: 03Dec2O13 Page 1 of 1

Issuing Authority:

Pace Virginia, Minnesota Quality Office

| Sample Condition Upon Receipt Client Name: | CP | | Project | #: [[| WO: | 1255278 |
|---|---------------------------------------|------------|-------------|--------------|-------------------------|---|
| Courier: Fed Ex UPS Commercial Pace | USPS Other: | Þ | llent | 1 | 25527 | |
| Tracking Number: | | | | 6.00 | | |
| Custody Seal on Cooler/Box Present? Yes | 40 | Seals Ir | ntact? | Yes | No | Optional: Proj. Due Date: Proj. Name: |
| Packing Material: Bubble Wrap Bubble Bag | s Nor | ie 🗀 | Other:_ | . | | Temp Blank? Yes No |
| Thermometer Used: 111336687 | Type of Ice | # / | Wet [|]Blue | □Non | e Samples on ice, cooling process has begun |
| Cooler Temp Read *C: 7.0 Cooler Temp Co Temp should be above freezing to 6*C Correction Factor | orrected °C: or: <u>0、3</u> | 3 | | d Initials | | ological Tissue Frozen? Yes No NA n Examining Contents: 10/14/15 Comments: |
| Chain of Custody Present? | Yes | □No | □N/A | 1. | | |
| Chain of Custody Filled Out? | Yes | No | □n/a | 2. | | |
| Chain of Custody Relinquished? | | □No | □N/A | 3 | | |
| Sampler Name and Signature on COC? | Yes | □No | □n/a | 4. | **** | |
| Samples Arrived within Hold Time? | ∠Yes | □No | □N/A | 5. | | |
| Short Hold Time Analysis (<72 hr)? | | No. | □n/a | 6. | | |
| Rush Turn Around Time Requested? | | No | □N/A | 7. | | |
| Sufficient Volume? | Yes | No | □n/a | 8. | | |
| Correct Containers Used? | √ZYes | ∏No | □N/A | 9. | | • |
| -Pace Containers Used? | Yes | □No | □N/A | | ~ | |
| Containers Intact? | Yes | □No | □N/A | 10. | | |
| Filtered Volume Received for Dissolved Tests? | Yes | 140 | ∐]N/A | 11. | | |
| Sample Labels Match COC? -includes Date/Time/ID/Analysis Matrix: | √Yes . | □No | □N/A | 12. | | |
| All containers needing acid/base preservation will be checked and documented in the pH logbook. | Yes | □No | ZIN/A | | oH log f menta | for results and additional preservation tion |
| Headspace In Methyl Mercury Container | □Yes | □No | ØN/A_ | 13. | | |
| Headspace In VOA Vials (>6mm)? | Yes | □No | DN/A | 14. | | |
| Trip Blank Present? | Yes | □No | □N/A | 15. | | |
| Trip Blank Custody Seals Present? | Yes | □No | N/A | | | • |
| Pace Trip Blank Lot # (if purchased): | | | | <u> </u> | | |
| CLIENT NOTIFICATION/RESOLUTION | | | | | | Field Data Required? Yes No |
| Person Contacted: | | | | Date/Tin | ne: | |
| Comments/Resolution: | | | | | | |
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| FECAL WAIVER ON FILE Y N | | TEM | PERATU | IRE WA | IVER OI | N FILE Y N |
| Project Manager Review: Walter S Note: Whenever there is a discrepancy affecting North Carolina | compliance s | amples, | a copy of t | his form | Date: _ will be sent | 10 115 15 t to the North Carolina DEHNR Certification Office (i.e. out of |